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FINAL REPORT

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NASW-96008

DEVELOPMENT OF A FOCUSSING HARD X-RAY TELESCOPE

PERIOD OF PERFORMANCE:
June 17, 1996 to June 16, 1997

PREPARED FOR:
NASA Office of Space Science
The Sun-Earth Connection
Solar Physics Supporting Research and Technology Program

PREPARED BY:
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SUMMARY

This document is the final technical report for contract NASW-96008, "Development of a Focussing Hard X-ray Telescope". The period of performance of the contract was from June 17, 1996 to June 16, 1997.

This contract was the most recent phase of an ongoing program at Radiation Science supported by NASA. The overall objective of this program is the construction of a focussing hard X-ray telescope.

The tasks to be accomplished under this contract included:

1. verify by optical measurement that CN coatings reduce the surface roughness of highly polished silicon carbide and sapphire optical surfaces;
2. generate electroformed nickel replicas of these master surfaces;
3. evaluate the quality by optical measurements and X-ray scattering tests of both: (a) the CN coated masters, and (b) the replicas electroformed on the masters;
4. deposit multilayers on *both* the replicas and a master which has been coated with a release agent;
5. measure the quality of the multilayer surfaces by optical and X-ray tests;
6. (a) generate a nickel replica of the multilayer coated master, (b) separate the multilayer from the master *between* the master surface and the multilayer; and,
7. measure the quality of the multilayer coated replica produced in task 6.

All of these tasks were completed successfully. Tasks 1 through 6(a) were completed within the period of performance of the contract. Tasks 6(b) and 7 were accomplished after the termination date of this contract by our collaborators at Northwestern University with the assistance of Radiation Science personnel at no cost to this contract.

We were able to generate replicated surfaces with arc-second range hard X-ray scattering properties. We were able to reflect 15, 30, and 45 keV X-rays from multilayer coatings on thin foils. We were able to separate a multilayer coating deposited directly on a master surface from the surface after electroforming a supporting structure without damaging either the multilayer or the master surface. These developments open the door for the fabrication of a Wolter type I hard X-ray telescope. We conclude that the development of such a telescope should proceed.

INTRODUCTION

This document is the final technical report for contract NASW-96008, "Development of a Focussing Hard X-ray Telescope". Submission of this report has been delayed because the final technical task in the Statement of Work was completed after the expiration of the period of performance. This contract was the most recent phase of an ongoing program at Radiation Science supported by NASA. The overall objective of this program is the construction of a focussing hard X-ray telescope having the spatial resolution and sensitivity to perform useful measurements of solar flares and micro-flares. The objective of this contract was to verify that a Wolter type I grazing incidence telescope for high resolution, hard X-ray imaging of solar flares can be manufactured within the present state of the art.

It is now well known that hard X-rays can be focussed by using diffraction phenomena to deflect their optical paths. The most versatile way to do this is to use multilayer diffractors in grazing incidence. One can use many of the techniques developed for focussing soft X-rays with grazing incidence optics. Several groups are pursuing this effort for celestial X-ray astronomy. At this time, the construction of a multilayer coated Kirkpatrick-Baez (KB) style hard X-ray telescope is within the state of the art. The requirements of solar flare physics would not be satisfied by a KB device. The Wolter style optics which previously dominated high resolution soft X-ray solar imaging, have higher resolution, greater geometrical collecting area for a given focal length, and higher efficiency than KB optics.

There were potential practical difficulties which we have investigated as preparation for the design of a Wolter I hard X-ray telescope for solar flares. In order to maximize the collecting area of such a telescope, it is necessary to nest coaxial reflecting elements as closely as possible. This implies that the figured substrates upon which the multilayers are deposited should be as thin as possible. In general, the thickness of the walls of a Wolter telescope is governed by the necessity to withstand polishing loads. This thickness is much greater than that required to maintain the figure of the telescope in a micro-gravity environment. The wall thickness can be minimized by decoupling polishing from the flight optics, i.e. the substrates for the flight optics should be *replicas* of the polished surfaces. This requires a significant advance in the surface smoothness of replica optics for two reasons. It is obviously necessary to minimize the intensity and angular spread of the radiation scattered from the reflecting surface. In addition, the reflectivity of a multilayer coating is a function of the smoothness of the surface on which the coating is deposited. This contract was devoted to investigating techniques for producing low scatter, multilayer coated, replicated optics.

One might think that to make such a telescope, we would have to deposit multilayers on the inside of the replicated mirrors after the mirrors are separated from the highly polished solid cylindrical mandrels from which they are replicated. There are several drawbacks to this approach. The replicated surfaces, on which the multilayers would be deposited are usually less smooth than the masters, and the need to accomodate a sputtering gun inside the optic sets a very serious limit on the minimum radius of the optic and hence, on the minimum focal length.

There is another technique that avoids these difficulties. In the work reported here, we determined that it is possible to deposit multilayer coatings on a master surface, electro-deposit the foil structure on top of the multilayer, and then separate the multilayer coated mirror from the master without destroying either the multilayer or the master surfaces. If this technique can be reproduced with cylindrical masters, it eliminates the problems associated with the inside deposition technique.

This project was performed in collaboration with Northwestern University. The group at Northwestern is among the world leaders in the production of low scatter replica optics. They provided test specimens under subcontract to Radiation Science. This work was an extension of efforts that the Northwestern group is performing under grant from the High Energy Astrophysics SR&T program. In addition, Dr. Peter Takacs of Brookhaven National Laboratory (BNL) measured the quality of some of the surfaces that we produced. His work was performed with no exchange of funds between Radiation Science and BNL.

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1. verify by optical measurement that CN coatings reduce the surface roughness of highly polished silicon carbide and sapphire optical surfaces;
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3. evaluate the quality by optical measurements and X-ray scattering tests of both: (a) the CN coated masters, and (b) the replicas electroformed on the masters;
4. deposit multilayers on *both* the replicas and a master which has been coated with a release agent;
5. measure the quality of the multilayer surfaces by optical and X-ray tests;
6. (a) generate a nickel replica of the multilayer coated master, (b) separate the multilayer from the master *between* the master surface and the multilayer; and,
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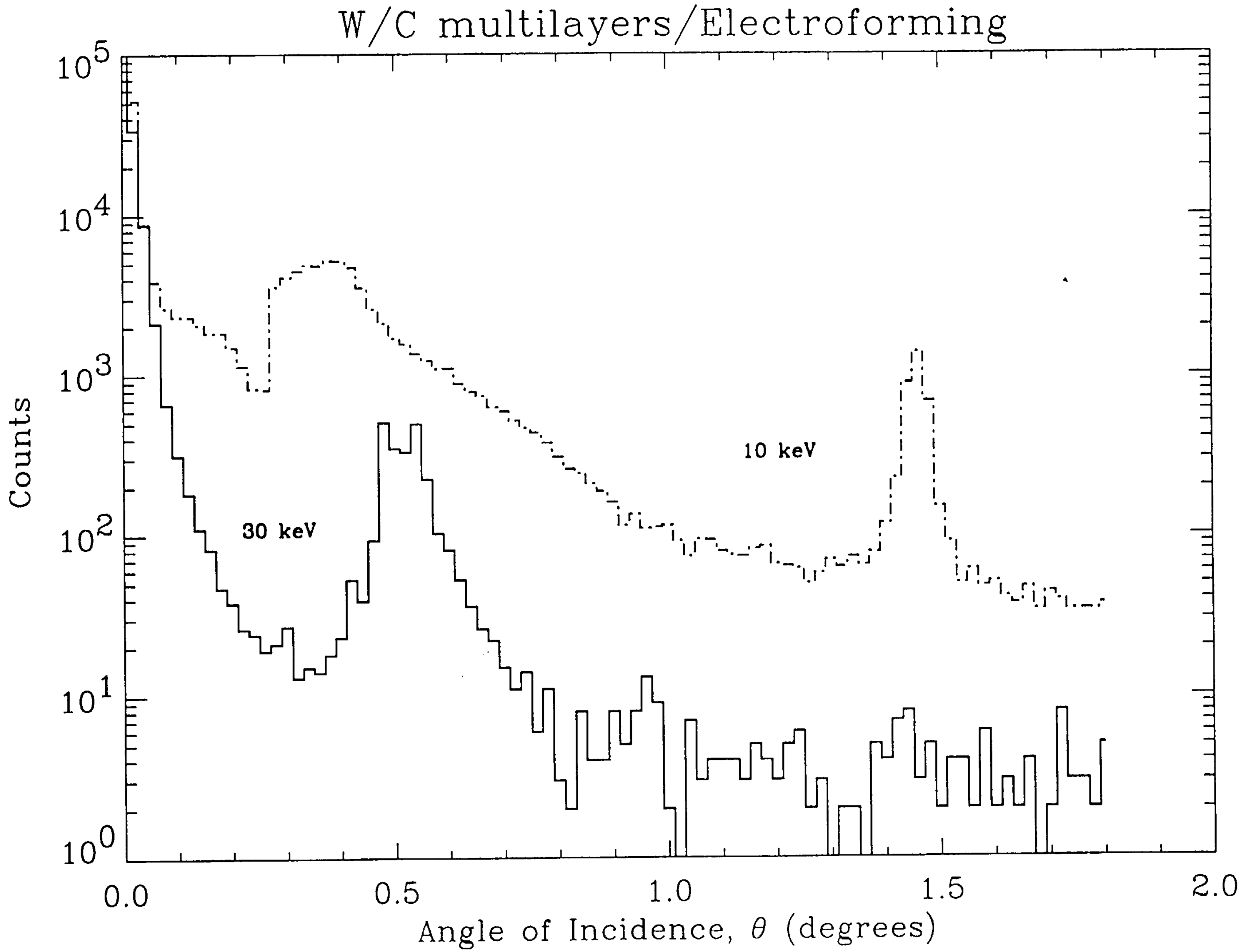
We measured the hard X-ray reflection efficiency and scattering width of the master surfaces, of electroformed replicas of these surfaces, and of multilayers deposited on both the masters and the electroforms. The X-ray measurements were made at the Advanced Photon Source at Argonne National Laboratory. Our final measurement proved the validity of our approach. Figure 1 (placed after the text) shows hard X-ray reflection at both 10 and 30 keV from a multilayer coated on a flat master surface and then separated from the master *after* the deposition of an electroformed support structure on top of the multilayer. This figure demonstrates the success of this program. These results were presented at the June, 1997 AAS meeting and at the July 1997 SPIE meeting.

If these results hold true for cylindrically symmetric surfaces, we will be able to deposit a depth graded multilayer diffracting film on the *outside* surface of a very smooth solid cylindrically symmetric mandrel instead of on a replica of that surface. In addition to generating a smoother surface, this technique has other advantages. It should be easier to deposit uniform multilayer coatings on the outside of a cylinder than it is to deposit them on the inside of the cylinder. In addition, the minimum radius of the telescope elements will not be constrained by the requirement to fit a sputtering mechanism inside the cylindrical telescope. This means that we can use a shorter focal length for our mirrors thereby reducing the size, weight and moment of inertia of the telescope.

Progress reports have been submitted covering the first three quarters of the contract. Those progress reports are attached to this document. The work conducted during the fourth quarter of the contract, and subsequently, was reported at the AAS and SPIE meetings. Written texts of the SPIE presentations are attached to this document.


W/C multilayers/Electroforming
Counts
10^5
10^4
10^3
10^2
10^1
10^0
30 keV
10 keV
0.0
0.5
1.0
1.5
2.0
Angle of Incidence, θ (degrees)

Quarterly Progress Report
NASW-96008
17 June 1996 to 16 September 1996

DEVELOPMENT OF A FOCUSSING HARD X-RAY TELESCOPE

Radiation Science, Inc.
P. O. Box 293
Belmont, MA 02178-0002

This report covers progress during the first quarter of this contract. Much of the work during this period was devoted to initiating the collaboration between Radiation Science, Inc. and Northwestern University. In addition, effort was devoted to an examination of Radiation Science's multilayer design and analysis code.

INTRODUCTION

The overall objective of this contract is to verify that a Wolter type I grazing incidence telescope for hard X-rays from solar flares can be manufactured within the present state of the art. It is now well known that hard X-rays can be focussed by using diffraction phenomena to bend their optical paths. The most versatile way to do this is to use multilayer diffractors in grazing incidence. One can use many of the techniques developed for focussing soft X-rays with grazing incidence optics. At this time, the construction of a multilayer coated Kirkpatrick-Baez (KB) style telescope is within the state of the art. Several groups are pursuing this effort for celestial X-ray astronomy. The requirements of solar flare physics would not be satisfied by these devices. The Wolter optics which previously dominated high resolution soft X-ray solar imaging, have higher resolution, greater geometrical collecting area for a given focal length, and higher efficiency than KB optics.

There are practical difficulties which we are investigating as preparation for the design of a Wolter I hard X-ray telescope for solar flares. In order to maximize the collecting area of such a telescope, it is necessary to nest coaxial reflecting elements as closely as possible. This implies that the figured substrates upon which the multilayers are deposited should be as thin as possible. In general, the thickness of the walls of a Wolter telescope is governed by the necessity to withstand polishing loads. This thickness is much greater than that required to maintain the figure of the telescope in a micro-gravity environment. The wall thickness can be minimized by decoupling polishing from the flight optics, i.e. the flight optics should be replicas of the polished surfaces. This requires a significant advance in the angular resolution of replica optics. It is necessary to reduce the intensity and angular spread of the radiation scattered from the reflecting surface. This project is devoted to investigating techniques for producing low scatter replicated optics.

PROJECT DEFINITION

This project is being performed in collaboration with Northwestern University. The group at Northwestern is among the world leaders in the production of low scatter replica optics. They will provide the test specimens under a subcontract with Radiation Science. This work will be an extension to the efforts that the Northwestern group will be performing under a grant from the High Energy Astrophysics SR&T program. In addition, Dr. Peter Takacs at Brookhaven National Laboratory has agreed to measure the quality of some of the surfaces that we will produce. His work will be performed with no exchange of funds between Radiation Science and BNL.

The initial task during this quarter was the development of specific objectives for this project, and the definition of the work to be performed by the various participants. The objectives include:

1. verify by optical measurement that CN coatings reduce the surface roughness of highly polished silicon carbide and sapphire optical surfaces;
2. generate electroformed nickel replicas of the these master surfaces;
3. evaluate the quality of both the CN coated masters and the replicas electroformed on the masters by optical measurements and X-ray scattering tests;
4. deposit multilayers on *both* the replicas and a master which has been coated with a release agent;
5. measure the quality of the multilayer surfaces by optical and X-ray tests;
6. generate a nickel replica of the multilayer coated master, and separate from the master *between* the master surface and the multilayer; and,
7. measure the quality of the multilayer (and release agent) coated replica produced in task 6.

REPLICATION

The group at Northwestern University has reactivated their electroforming setup. In addition, they have prepared to coat one inch diameter, highly polished, ceramic masters with CN. The surfaces of the CN masters will undergo optical inspection in the near future. X-ray tests of both the masters and electroformed replicas are scheduled for December and January.

MULTILAYER DESIGN

Northwestern and Radiation Science have discussed the characteristics of the multilayers and the release agent to be deposited on the masters.

It is necessary to coat the master surface prior to electroforming with a release agent which will facilitate separation of the replica from the master. Normally, the release agent would be gold. In this case, however, the release agent is not the X-ray reflecting surface. Instead, the X-rays will be reflected by the multilayer coating below (from the standpoint of an incoming X-ray) the release agent. The release agent should therefore be as transmissive as possible. We have tentatively agreed to try nickel.

We have also devoted some thought to the design of the multilayer coating, itself. The ideal multilayer coating for a hard X-ray telescope is a depth graded multilayer with longer period bilayers at the top to reflect low energy X-rays and shorter period bilayers below to reflect higher energy X-rays. In this case, however, we will be reflecting X-rays from *both* sides of the same multilayer coatings (in steps 5 and 7). Therefore, the multilayer coatings must be symmetric. There are two ways that this could be accomplished. We could use a constant period multilayer. This has the virtue of simplicity, but a constant period multilayer will reflect X-rays of a given energy at only one angle. Finding that angle, during X-ray testing may be time consuming. It also adds another source of uncertainty to the measurements. Consequently, we are also considering the design of a multilayer whose periods are symmetric about the center of the coating.

In addition, we have begun to reassess our choice of materials for the multilayer coating. In the past, we have compared the predicted X-ray reflectivity of W:Si multilayers with that of Ni:C multilayers. W:Si multilayers have always looked better than Ni:C in our analyses. A recent re-examination of the reflection data that we obtained with the same multilayer at 37 and 45 keV at Brookhaven National Laboratory has indicated the presence of an anomaly in the comparison of these results at constant $E\sin\theta$ with our models. This could be attributed to an error in the way that absorption is treated in our code. If so, we will reconsider the tradeoff between W:Si and Ni:C.

SECOND QUARTER PLAN

During the second quarter of the period of performance of this contract, 9/17/96 to 12/16/96, replication activity will include inspection of the CN coated master and the production of the initial replicas so that X-ray testing can begin at the start of the third quarter. Tests will be conducted to demonstrate stability of the CN coating with electroless nickel and in the liquid nitrogen used to separate the master from the replica. These will include both optical inspection and AFM microscopy in order to characterize surface roughness at the spatial frequencies that scatter X-rays. The initial replicas will be produced after these tests. The replicas will also be examined both by Nomarski and by AFM microscopy.

At this writing, a review of the treatment of absorption in the Radiation Science multilayer reflectivity code is in progress. If necessary, we will modify the code and reassess the multilayer materials prior to the design of the multilayer. We believe that the multilayer coating design will still be available prior to multilayer deposition in the third quarter of the contract. In addition, we have commenced talks with the operators of the synchrotron beam line at Argonne National Laboratory that we intend to use for the X-ray tests. We have found this to be an important means of saving time and effort at the synchrotron.

At this time the program appears to be on schedule. By the end of the second quarter, we expect to have finished the first three tasks in our program plan with tasks four and five in progress. There may be some additional, unbudgeted, effort in design of the multilayer coatings, but this should be accommodated without perturbing the schedule and within the available resources.

Quarterly Progress Report
NASW-96008
17 September 1996 to 16 December 1996

DEVELOPMENT OF A FOCUSSING HARD X-RAY TELESCOPE

Radiation Science, Inc.
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This report covers progress during the second quarter of this contract. Much of the work during this period was devoted to preparing the facilities at Northwestern University for the production of the smooth masters for measurement and multilayer deposition. Effort at Radiation Science was hindered by the loss of the services of the principal investigator for the final month of this quarter due to illness. Prior to his illness the PI completed the modifications to the multilayer analysis code required for accurate results at energies where the absorption term in the index of refraction is significant.

INTRODUCTION

The overall objective of this contract is to verify that a Wolter type I grazing incidence telescope for hard X-rays from solar flares can be manufactured within the present state of the art. It is now well known that hard X-rays can be focussed by using diffraction phenomena to bend their optical paths. The most versatile way to do this is to use multilayer diffractors in grazing incidence. One can use many of the techniques developed for focussing soft X-rays with grazing incidence optics. At this time, the construction of a multilayer coated Kirkpatrick-Baez (KB) style telescope is within the state of the art. Several groups are pursuing this effort for celestial X-ray astronomy. The requirements of solar flare physics would not be satisfied by these devices. The Wolter optics which previously dominated high resolution soft X-ray solar imaging, have higher resolution, greater geometrical collecting area for a given focal length, and higher efficiency than KB optics.

There are practical difficulties which we are investigating as preparation for the design of a Wolter I hard X-ray telescope for solar flares. In order to maximize the collecting area of such a telescope, it is necessary to nest coaxial reflecting elements as closely as possible. This implies that the figured substrates upon which the multilayers are deposited should be as thin as possible. In general, the thickness of the walls of a Wolter telescope is governed by the necessity to withstand polishing loads. This thickness is much greater than that required to maintain the figure of the telescope in a micro-gravity environment. The wall thickness can be minimized by decoupling polishing from the flight optics, i.e. the flight optics should be replicas of the polished surfaces. This requires a significant advance in the angular resolution of replica optics. It is necessary to reduce the intensity and angular spread of the radiation scattered from the reflecting surface. This project is devoted to investigating techniques for producing low scatter replicated optics.

This project is being performed in collaboration with Northwestern University. The group at Northwestern is among the world leaders in the production of low scatter replica optics. They will provide the test specimens under a subcontract with Radiation Science. This work will be an extension to the efforts that the Northwestern group will be performing under a grant from the High Energy Astrophysics SR&T program. In addition, Dr. Peter Takacs at Brookhaven National Laboratory has agreed to measure the quality of some of the surfaces that we will produce. His work will be performed with no exchange of funds between Radiation Science and BNL.

The tasks to be accomplished under this contract include:

1. verify by optical measurement that CN coatings reduce the surface roughness of highly polished silicon carbide and sapphire optical surfaces;
2. generate electroformed nickel replicas of the these master surfaces;
3. evaluate the quality of both the CN coated masters and the replicas electroformed on the masters by optical measurements and X-ray scattering tests;
4. deposit multilayers on *both* the replicas and a master which has been coated with a release agent;
5. measure the quality of the multilayer surfaces by optical and X-ray tests;
6. generate a nickel replica of the multilayer coated master, and separate from the master *between* the master surface and the multilayer; and,
7. measure the quality of the multilayer (and release agent) coated replica produced in task 6.

Task 1 is complete. Tasks 2 and 3 are in progress. The design of the multilayers to be deposited in task 4 is also in progress.

REPLICATION

During the previous quarter, the group at Northwestern University reactivated their electroforming setup. Unfortunately, the actual use of the system was delayed by the need to obtain certification of the safety of the system by the university. The certification was obtained at the start of the third quarter.

Prior to replication, the highly polished ceramic masters are coated with ZrN and/or CN. This coating is expected to smooth the surface of the master and to make the surface more durable. The first ceramic masters to undergo this treatment were examined both optically and by Atomic Force Microscope at Brookhaven National Laboratory (BNL). The results were encouraging. The AFM demonstrated that the coating had reduced the amplitude of the high spatial frequency (freq. $> 10\ \mu m^{-1}$) fluctuations in the surface and had probably reduced the amplitudes and increased the slope at intermediate frequencies ($10^{-1} <$ freq. $< 10\ \mu m^{-1}$).

The X-ray measurements were also initiated during this quarter. The masters that had been scanned at BNL were taken to the Advanced Photon Source at Argonne

National Laboratory. Because of illness, the Principal Investigator was unable to participate in this trip. Radiation Science's experienced consultant for such measurements was also unavailable. As a consequence, the setup of the experiment might have taken longer than it would have if the PI or our consultant had been present. The X-ray reflectivity of the masters was measured, but scattering profiles were not obtained. The participants from Northwestern and from the beam line gained valuable experience which will facilitate the next round of measurements.

It is necessary to coat the master surface with a release agent which will facilitate separation of the replica from the master. Normally, the release agent would be gold. In this case, however, the release agent is not the X-ray reflecting surface. Instead, the X-rays will be reflected by the multilayer coating below (from the standpoint of an incoming X-ray) the release agent. The reflected X-rays will pass through the release agent twice. The release agent should therefore be as transmissive as possible. We decided to use an evaporated layer of nickel as the release agent. This required a modification to the "boat" used to hold the material to be evaporated. The modification was completed during this period.

MULTILAYER DESIGN

During the first quarter of this program, We devoted some thought to the design of the multilayer coating, itself. The ideal multilayer coating for a hard X-ray telescope is a depth graded multilayer with longer period bilayers at the top to reflect low energy X-rays and shorter period bilayers below to reflect higher energy X-rays. In this case, however, we will be reflecting X-rays from *both* sides of the same multilayer coatings (in tasks 5 and 7, respectively). Therefore, the multilayer coatings must be symmetric. There are two ways that this could be accomplished. We could use a constant period multilayer. This has the virtue of simplicity, but a constant period multilayer will reflect X-rays of a given energy at only one angle. Finding that angle, during the setup phase of the X-ray testing may be time consuming. It also adds another source of uncertainty to the measurements. Consequently, we are also considering the design of a depth graded multilayer whose periods are symmetric about the center of the coating.

In addition, we have begun to reassess our choice of materials for the multilayer coating. In the past, we have compared the predicted X-ray reflectivity of W:Si multilayers with that of Ni:C multilayers. W:Si multilayers have always looked better than Ni:C in our analyses. A re-examination of the reflection data that we obtained at Brookhaven National Laboratory indicated the possibility of an error in the way that absorption is treated in our code. During this quarter we determined that the error was not significant at 37 or 45 keV. Because these mirrors will be tested at energies as low as 8 keV, the multilayer reflectivity code was modified and the modification was tested. Comparison of predicted reflectivities for W:Si multilayers and Ni:C multilayers was initiated but not completed.

THIRD QUARTER PLAN

During the third quarter of the period of performance of this contract, 12/17/96 to 3/16/97, replication activity will include the production of the initial replicas so that their X-ray testing can begin in the third quarter. The tests will include both optical inspection and AFM microscopy in order to characterize surface roughness at the spatial frequencies that scatter X-rays.

We will use the modified multilayer reflection code to reassess the multilayer materials prior to the design of the multilayer. The multilayer coating design will still be available prior to multilayer deposition.

At this time the program appears to be approximately three weeks behind schedule. By the end of the third quarter, we expect to have finished the first three tasks in our program plan with tasks four and five in progress. There will be some additional, unbudgeted, effort in design of the multilayer coatings, but this should be accommodated without perturbing the schedule and within the available resources.

Quarterly Progress Report
NASW-96008
17 December 1996 to 16 March 1997
Revised 29 June 1997

DEVELOPMENT OF A FOCUSSING HARD X-RAY TELESCOPE

Radiation Science, Inc.
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This report covers progress during the third quarter of this contract. Work during this period was devoted to the production of smooth master surfaces and surface quality measurements prior to multilayer deposition. In addition to AFM and optical measurements, we made X-ray scattering measurements at the Advanced Photon Source at 15 and 45 keV of one master surface. The X-ray measurements are under analysis at this time. We completed oui analytical investigation of the differences in the reflection profiles of Ni:C and W:Si multilayers at various energies. This led to the selection of a W:Si multilayer for our X-ray tests. We also completed our measurements of the adhesion of multilayer coatings to electroformed surfaces. We are preparing a paper for the annual SPIE meeting on X-ray optics, describing our technique for producing multilayer coatings on the insides of cylindrical elements. In addition, the results of a comparison between the optical and X-ray scattering measurements and their implications will be presented in a paper at the SPIE meeting.

INTRODUCTION

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6. generate a nickel replica of the multilayer coated master, and separate from the master *between* the master surface and the multilayer; and,
7. measure the quality of the multilayer (and release agent) coated replica produced in task 6.

Task 1 is complete. Task 3A, the characterization of the master surfaces is complete. Tasks 2, 3B, and 4 are in progress. Tasks 5 and 6 have been scheduled for the fourth quarter.

REPLICATION STUDIES

During the first quarter of this contract, the group at Northwestern University reactivated their electroforming setup. Unfortunately, the actual use of the system was delayed by the need to obtain certification of the safety of the system. The certification was obtained at the start of this quarter. Work this quarter has been devoted to the optimization of the replication process. This work was completed at the beginning of the fourth quarter and replication of the smooth surfaces has begun.

X-RAY SCATTERING MEASUREMENTS

Successful X-ray measurements of surface scatter from a smooth master which had been characterized at BNL were accomplished during this quarter. The masters that had been scanned at BNL were taken to the DND-CAT beamline of the Advanced Photon Source at Argonne National Laboratory. The beamline monochromator provided a 15 keV beam with a significant amount of energy at 45 keV and some energy at 30 keV. An adjustable width tantulum slit was used to define the input beam. It was set to its minimum width of about 2 microns. The sample height could be adjusted with an indicated precision (NOT accuracy) of one micron. The sample rotation angle could be adjusted with an indicated precision of 0.001 degree. The detector position was independent of the sample angle. Another adjustable slit set was used as the detector aperture. Although the width of this slit could be set to an indicated precision of 0.5 microns, irregularities in the edges of the slit jaws and uncertainty in the parallelism of the two jaws reduced the actual precision of the slit width an estimated value of one micron. The height of the detector aperture, was controlled with an indicated precision of one micron. The horizontal distance between the sample rotation axis and the detector slit was 3,581.5 $\pm$ 0.5 mm. If the accuracy of all the position measuring systems had been equal to their indicated precisions, this would imply a resolution in the scattering profile of 0.06 seconds of arc. Of course, this was not the case. We estimate that the actual limiting resolution of scattering profiles measured with this system would be a few tenths of an arc second.

A NaI scintillator attached to a single channel discriminator allowed us to distinguish the 15 keV beam from the higher harmonics. We measured the profile of the scattering around the specularly reflected beam at both 15 and 45 keV at grazing angles below the respective critical angles for total external reflection. The 30 keV beam (which shouldn't exist at all, theoretically) was too weak for measurement during the time allotted.

If the spatial frequency spectrum of a specularly reflecting surface is known, it should be possible to predict the scattering profile of that surface as a function of the energy of the reflected radiation. Previous attempts to do this have not been completely successful. This has been attributed to incomplete knowledge of the profile of the scattering surface. Because of the general interest in X-ray scattering from highly polished surfaces for astronomy, materials science, synchrotron radiation studies, and X-ray lithography, our surfaces have been characterized as well as possible at this time by expert metrologists. The very short wavelength, and the high precision of our measurements, make this a unique test of scattering theory. These results will be presented at the July 1997, annual meeting of SPIE.

RELEASE TESTS

It is necessary to coat the master surface with a release agent to facilitate separation of the replica from the master. For soft X-ray grazing incidence optics, the release agent is a 500 Å layer of gold evaporated on the surface of the mandrel. After separating the replica from the mandrel, the gold layer becomes the X-ray reflecting surface. In this case, however, the release agent is not the X-ray reflecting surface. Instead, the X-rays will be reflected by the multilayer coating below (from the standpoint of an incoming X-ray) the release agent. The reflected X-rays will pass through the release agent twice. The release agent should therefore be as transmissive as possible.

We decided to use an evaporated layer of nickel as the release agent. W:Si multilayers, only a few layers thick, were deposited on small (about 2 cm) masters which had been coated with either an evaporated gold layer, or an evaporated nickel layer. Some of the substrates for the nickel evaporation run had been held at high temperature during evaporation, while others had been kept cool. We found that the multilayers adhered better to the nickel which had been evaporated on the cool substrate than on the hot substrate samples. This might be explained, if we assume that the nickel on the uncooled substrate reached its fusion temperature and then crystallized when it cooled, while the nickel deposited on the cooled substrate never reached its fusion temperature and therefore remained amorphous.

MULTILAYER DESIGN

The ideal multilayer coating for a hard X-ray telescope is a depth graded multilayer with longer period bilayers at the top to reflect lower energy X-rays and shorter period bilayers below to reflect higher energy X-rays. In this case, however, we will be reflecting X-rays from *both* sides of the same multilayer coatings (in tasks 5 and 7, respectively). Therefore, the multilayer coatings must be symmetric. There are two ways that this could be accomplished. We could use a constant period multilayer. This has the virtue of simplicity, but a constant period multilayer will reflect X-rays of a given energy at only one angle. Finding that angle, during the setup phase of X-ray testing at a synchrotron beamline can be time consuming. It also adds another source of uncertainty to the measurements. Consequently, we also considered the design of a depth graded multilayer whose periods are symmetric about the center of the coating. But, in this case, the X-rays are actually reflected by two distinct multilayers, which would differ because of the (± 1 or 2% per layer) imprecision of the deposition process. We ultimately concluded that our results would more accurately demonstrate the effect of replication and separation if we used a constant period multilayer with a 500 Angstrom nickel coating on either side.

In addition, we have begun to reassess our choice of materials for the multilayer coating. In the past, we have compared the predicted X-ray reflectivity of W:Si multilayers with that of Ni:C multilayers. W:Si multilayers have always looked better than Ni:C in our analyses. A re-examination of the reflection data that we had obtained at Brookhaven National Laboratory under our previous contract indicated the possibility of an error in the way that absorption was treated in our multilayer analysis code. During this quarter we determined that the error was not significant at 37 or 45 keV, where we had previously measured, but that absorption might be significant at lower energies. We had originally intended to measure these mirrors at energies as low as 8 keV. The multilayer reflectivity code was modified. Comparison of predicted reflectivities for W:Si multilayers and Ni:C multilayers indicated that absorption could be neglected at energies above about 20 keV. Our present plan calls for measurements of the multilayer coated surfaces at 25 keV. Accordingly, we have ordered W:Si multilayers manufactured with the same techniques we have used previously.

FOURTH QUARTER PLAN

During the fourth quarter of the period of performance of this contract, 3/17/96 to 6/16/97, replication of *some* of the masters will be completed. The replica surfaces will be characterized at Brookhaven using the same techniques that were used on the master surfaces. The tests will include both optical interferometry and AFM microscopy in order to characterize surface roughness at the spatial frequencies that scatter X-rays. Multilayer coatings will be deposited on the replicas and on those master surfaces which have not been replicated.

We are presently scheduled to perform X-ray scattering tests at Argonne National Laboratory between June 2 and June 14. This run will be different from our previous synchrotron experiences in that we will have use of the beam for only 8 to 12 hours per day for two weeks. Our experimental setup will remain in place for the entire period and the beam will be intercepted in another hutch closer to the synchrotron than ours, when we are not observing. In all our previous measurements at synchrotrons, we have had four or five days to occupy the hutch, set up our experiment, align it to the beam, make our measurements, and get out. The new arrangement will allow us to use our beam time much more efficiently.

PROGRAM STATUS

At this time the program appears to be six to eight weeks behind schedule. At the present contract termination date of June 16, 1997, tasks 6 and 7, the replication and testing of the multilayers deposited on the masters, will not be complete. These tasks will be carried out under a different program. After X-ray testing of the multilayer coated masters at Argonne National Laboratory, they will be returned to Northwestern for the electroforming of support surfaces on top of the multilayer coatings. The multilayers and their supports will then be separated from the masters. These multilayer coated "replicas", will then be examined both optically and in X-rays during the summer and autumn of 1997, under a subcontract to Radiation Science from Northwestern University under a grant from the NASA High Energy Astrophysics Branch at no further cost to this contract.

Grazing incidence and multilayer X-ray optical systems

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ABSTRACT

The development of X-ray optics for astrophysical investigations in the 40–100 keV energy range is extremely important. In this energy range, a focusing system is necessary to resolve crowded regions, to improve sensitivity, and to provide the deep sky images necessary to make the next great step forward in this field. Such a step was ably demonstrated by the Einstein and ROSAT observatories. These systems used grazing incidence optics, and, as is well known, the critical angle of reflectivity decreases linearly with energy for ordinary metal surfaces which adversely impacts on the design of a focusing system for higher energy X-rays. At least 3 parameters are negatively affected: (a) the field of view is decreased; (b) the projected area of an individual mirror element is decreased; and, (c) the focal length for a fixed diameter system is increased. In order to counter these effects, mirrors coated with multilayers have been designed. It is theoretically possible to increase the grazing angle by coating the mirror surface with a graded d-spacing. The ability to produce a coated mirror with close to theoretical performance is, however, technically challenging. We describe our approach to the fabrication of a system designed for the 40–100 keV range that is based on electroforming technology. We also describe some of the general considerations that must be taken into account when fabricating a viable mirror.

Keywords X-rays, Multilayers, Wolter I, Coatings

1. INTRODUCTION

The motivations for developing Wolter I-type optics with multilayer spacing are many. For astronomical purposes the multilayers allow us to make studies that are not possible without a focusing system. For example, crowded regions such as the galactic center demand focusing

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optics, and so do deep sky observations with high sensitivity. Imaging of non-thermal X-ray sources such as supernova remnants also needs the combination of resolution and sensitivity that only focusing optics can provide. See also refs. 1 & 2 for discussions of the science that can be done with focusing optics in hard (40–100 keV) X-rays.

The first issue to address was designing the multilayers and testing them by themselves. Work by Joensen et al. (ref. 3, and references therein), Christensen et al.[4], and Krieger et al.[5] has demonstrated that graded d-spacing multilayers are capable of providing substantial improvements in the high energy reflectivity of X-ray mirrors.

The key requirement of the astrophysics is that the multilayers be placed on the inside of a Wolter I optic and that this should be between 2 and about 20 cm in diameter. These diameters are too small to deposit multilayers via typical sputtering techniques. In order to meet this requirement, we need an innovative fabrication technique, which is electroforming. Before the electroforming is done, multilayers are deposited on an electroforming master, and, as long as a proper buffer/release agent is on the master, the multilayers will release from the master and stick to the electroform. This method has been successfully used by our group and others to produce nickel mirrors that are coated with gold on the inside. Note that, although there are other possible geometries to use for X-ray optics besides the Wolter I type (cf. ref. 3, 4), the advantage of using such a system is that it provides the best chance of producing an angular resolution of better than 1 arc-minute.

The basic requirements of an X-ray optic are: (1) the surface must be smooth on a 3Å scale so as to minimize scattering effects; (2) the graze angle at which reflection takes place must be as large as possible to optimize the field of view and collecting area; (3) the finished product should be an optic that can be aligned and mounted as easily as possible; and, (4) the production costs of the individual X-ray reflecting segments (or mirrors) must be as low as possible, as it is likely that deep nesting and/or many modules will be required (at least for astronomical purposes).

The basic material requirements for the replication process are: (1) the mandrels should have as smooth a surface as possible, i.e., in the 3Å range; (2) the surface must be durable so as to allow for multiple copies; (3) the surface of the mandrel should allow for the uniform deposition of the multilayers, yet these surfaces must release the multilayers when the replicated mirror is removed; (4) the multilayers must adhere to the mirror surface; (5) the multilayers must remain intact throughout the entire production process; and, (6) the stresses built up by the multilayer deposition should not measurably deform the mirror.

As we demonstrate below, we believe we have an approach which can address all of the requirements successfully. However, we have alternative methods and approaches we will follow, if necessary, so that the end result of this project will be to have established the viability of a technology for producing Wolter I-like 40–100 keV optics.

2. REVIEW OF PREVIOUS WORK

As noted above, Joensen et al.[3] have discussed recent work done to study the fabrication of multilayer-coated mirrors. They had several concerns: (1) the smoothness of the substrate on the nano-meter scale; (2) the deformation of the mirror due to stresses built up by the multilayers; (3) the use of materials other than nickel (or steel) for the electroform master surfaces; (4) the difficulty of electroforming flats; and, (5) the use of a buffer agent on the electroformed master surface.

We are using techniques that will smooth the master on the nano-meter scale down to 1.5Å (see CN_x-coating discussion below). For the master flats, on the micrometer scale the roughness should be below 2Å, and currently we have electroformed gold-coated flats that on the micrometer scale are as smooth as 3Å. These numbers compare favorably with those produced by Joensen et al.[3] (10–30Å for the nano-meter scale and 6–13Å for substrates on the micrometer scale).

The deformation of the mirror due to stresses is handled by choosing the materials so that differences in CTE are minimized. It should be noted that the NiC layers used by Joensen et al.[3] did not produce a prohibitively high stress as long as the sputtering rate was controlled.

Materials other than nickel have been used successfully. We have replicated our ultra-smooth (<3Å rms roughness) electroforms from materials such as sapphire and CVD SiC[6]. Furthermore, we have demonstrated that Ni has approximately the same adhesion to our electroform master surface (CN_x) as gold does to the sapphire and CVD SiC and, therefore, we anticipate being able to fabricate extremely smooth multilayer-coated replicas. Producing electroformed flats has not been a problem for us, as we have made over 2 cm pieces flat to at least a quarter of a wave.

Finally, we note that Joensen et al.[3] unsuccessfully used a carbon coating on the electroformed master both for smoothing and as a release agent. Based on previous CN_x-coating work (described below), and from successful electroforming using sapphire and CVD SiC, we are confident that we have found a good buffer/smooth coating in CN_x that will provide the proper release in the electroforming process. Also, work at Northwestern explicitly compared at least some carbon coatings with our CN_x coatings, and demonstrated that the CN_x coatings are indeed smoother.

Both Joensen et al.[3] and White et al.[2] have remarked that NiC (versus SiW) is necessary to make multilayers for use in the energy range above about 69 keV. As was shown by Krieger et al.[5], however, it is possible to design multilayers with tungsten that works above the tungsten K-edge. The d-spacing design is different from that used to obtain optimal reflectivity below 69 keV. But as these coatings would only be used on the inner mirrors of a nested set, the design does not affect the overall performance of the mirror set. Therefore, it is possible to design a multilayer-coated mirror system that operates out to 100 keV but all the mirrors are coated with SiW.

3. TECHNICAL APPROACH

3.1. Overview

It has been clear for many years that there was a requirement in X-ray astronomy for a focusing system that worked to as high an energy as possible. After a great deal of thought as to the best method of filling this demand, the consensus seems to be that Wolter I optics is to be preferred over both Baez-Kirkpatrick and conical foil designs (cf. ref. 3; also ref. 7; but see White et al.[2], who propose the possibility of using foils). Thus, here we only consider the fabrication of Wolter I optics.

We would like to produce multilayer-coated electroformed nickel mirrors capable of reflecting 40–100 keV X-rays using carbon nitride-coated electroless nickel/aluminum masters. The substrate material for the mandrel was chosen for two reasons: (1) the electroless-nickel/aluminum combination is an industry standard from which ultra-smooth mandrels are fabricated at reasonable cost in a variety of shapes (flats, spheres, Wolter I, etc.); and, (2) aluminum has a high coefficient of thermal expansion which enables replica removal through differential contraction in liquid nitrogen. Carbon nitride is a relatively new material whose advantages in this application are threefold: (1) it has a hardness nearly equal to that of diamond and will therefore protect masters through multiple replication operations and virtually eliminate the need for repolishing; (2) it can reduce the high-frequency roughness of the underlying substrate; and, (3) it provides a surface that has the necessary adhesion properties for the multilayer/electroforming process. Below we describe the main technologies and design issues in more detail.

3.1.1. Power Law Multilayers In the design of visible light multilayers and neutron super mirrors, if absorption is neglected, and if the number of layers in a multilayer becomes very large, d_i, the period of the ith layer, is equal to $d_C i^{-0.25}$, where d_C is the period corresponding to the critical energy at the angle of the multilayer. In cases where i is not large and absorption is not negligible, this formula must be modified, but it remains a good place to start for a multi-parameter design.

Joensen et al.[8] have used the formula $d_i = a(b-i)^{-c}$. They obtained their best results with $c = 0.27$. In all cases, they used equal thicknesses of both materials in their bi-layers. Theoretical considerations (see ref. 9) indicate that, in cases involving absorption, better reflectivity is obtained for lesser thicknesses of the high-Z material. Radiation Science has designed and tested depth-graded multilayers for the range 20 to 70 keV[5] using this formula. The multilayers behaved as expected; showing more or less constant reflectivity (with oscillations) for energies less than the tungsten K-edge at 69.5 keV.

As an alternative approach, we considered families of multilayers satisfying the general formula above with values of the constant c between 0.25 and 0.30. Instead of using equal thicknesses of both the high-Z and low-Z materials in each bi-layer, we specified the thickness of the high-Z layer to be 9Å. This is the minimum thickness for uniform depositions of high-Z elements. For each family of multilayers, we specified a (varying) number of periods to be generated with thicknesses between d_{min} and d_{max}. We used values of d_{min} and d_{max} which extended over larger ranges than the range of equivalent constant periods for the minimum and maximum energies. The constants a and b were chosen to satisfy these conditions, and the multilayers were generated from the formula. As expected, we found that better reflectivity could be expected from multilayers designed with minimum W thickness and slightly larger values of the constant c.

3.1.2. Carbon Nitride Coatings Carbon nitride thin films are synthesized using dc unbalanced magnetron sputtering of a high-purity graphite target in a nitrogen-containing plasma onto various substrates kept at ambient temperatures. The N/C ratio was found to vary from 0 to 0.8 depending on deposition conditions. There was evidence of multiple bonding states for carbon and nitrogen (sp, sp^2, and sp^3) and the bulk of the film was found to be amorphous. Nano-indentation studies showed that under appropriate substrate-bias conditions, the carbon nitride coating hardness can be enhanced to levels (25–30 GPa) well above that of conventional amorphous hard carbon coatings. CN_x coatings replicate the substrate topography, giving rise to surface roughness equal to or lower than that of the original substrates. To demonstrate the durability of these coatings, we note that lubricated tribotesting showed the relationship between wear performance and deposition conditions. Under pure sliding in air, the CN_x coating provides a 30-fold reduction in wear rate for M2 steels (compared with uncoated M2) under our testing conditions. Pin-on-disk wear testing of ultra-smooth magnetic thin-film disks with 5 nm CN_x overcoats showed three- to four-fold improvements in contact durability compared to amorphous-carbon-coated disks.

We noted in our earlier investigations that CN_x coatings appear to replicate the substrate topography. In applications such as protective overcoats for computer hard disk surfaces, it is imperative that such overcoats be as smooth as possible. We sputter-etched away the original amorphous-carbon-coating from 95mm-diameter ultra-smooth nichrome-coated hard disks. Then using nichrome-coated disks as starting substrates, we deposited 5 nm-thick CN_x coatings under a total (argon + nitrogen) pressure of 4 mTorr, 0.2 mTorr nitrogen, 1.0 kW target power and -250 V pulsed dc substrate bias. We used AFM to measure the rms surface roughness σ over scan areas of 500 nm $\times$ 500 nm. The σ for amorphous-carbon-coated substrates was found to be 0.63 ± 0.05 nm, while that for CN_x-coated surfaces averaged 0.25 ± 0.05 nm.

The difference may be related to the use of nitrogen. During deposition, the growing film (carbon being the major component) is constantly bombarded by species in the plasma. Since the atomic mass of nitrogen is closer to that of carbon than that of argon, momentum transfer is more efficient with nitrogen. Therefore, any hillocks or rough features formed on the growing film can be more efficiently removed by nitrogen bombardment, which results in the smooth surface morphology.

The above was taken from published work[10], however, further work has shown that it is possible to reduce initial nichrome-coated surfaces from about 10Å (1 nm) to 1.5Å (0.15 nm) with the CN_x coating. Therefore, CN_x-coated substrates can be made extremely smooth, smoother than those coated with amorphous carbon. We will, therefore, use this technology to our advantage in this current project.

3.2. Replication Technology

The electroforming work is performed by the electroform optics group at Northwestern University. This system consists, in part, of a 380-L polypropylene electroplating tank, a 38-L auxiliary tank used for continuous low voltage electroplating (for purification and stress reduction), and a 420-L tank used for treatment and storage. The tanks are interconnected through transfer pumps and filters. A 5500 L/hr pump/filter system is used to continuously filter and agitate the bath. A 4500 L/hr in-tank filtration system is used for skimming, additional agitation and filtration, and occasional activated carbon treatment. An in-tank heater maintains the bath temperature at 125° F. The pH is continuously monitored and automatically controlled to 4.0 ± 0.05. An automatic filling system replaces water lost to evaporation.

To avoid excessive thickness at the edges and non-uniform stress, mandrels are held in mounts designed to minimize electric field variation and are generally rotated at 40 rpm. To avoid "burning" substrates with relatively thin conductive layers, current can gradually be increased over several minutes to that required for zero stress.

Flat replicas can generally be removed directly from masters. However, mirrors with cylindrical symmetry and shallow graze angle generally require differential contraction in liquid nitrogen. In the latter case, the mirror assembly is drained, rinsed, transferred to an insulated container, and chilled in liquid nitrogen. By turning a threaded shaft, the mirror is guided away from the mandrel. (The mirror does not rotate during this process; the shaft also prevents the mirror from scraping against the mandrel.) The mirror and mandrel are rinsed and blown dry. The mandrel is allowed to reach room temperature, solvent-cleaned, and prepared for next use.

3.3. Coating, Buffer Layers, and Replication

We have found that Ni works as a release agent from the CN_x coating, and we have plans to experiment with other materials as well.

Supposing that the surface smoothness of the curved surfaces of the mandrels made with electroless nickel over-coated with CN_x is just not good enough; there exist several possible solutions. First, we could use CVD SiC coating on metal substrates, and have the CVD SiC polished. If we were to use solid CVD SiC, these mandrels would be much more expensive than electroless nickel-coated Al mandrels, so we would like to avoid this approach, but it *is* another possibility. Yet another possibility is to use a 2 micron layer of CN_x and to have this surface polished. CN_x is nearly as hard as diamond and has already been shown to produce 1.5Å smoothness on the nano-meter length scale. Therefore we expect that polishing CN_x is another possible solution.

Our conclusion is that there are possible solutions to difficulties that might arise related to the coatings and buffer layers/release agents necessary to produce multilayers on the insides of mirrors.

4. SUMMARY AND CONCLUSIONS

As noted in the introduction, there are many astrophysical investigations that would benefit from the development of Wolter I-type optics made with multilayer coating. The concept is relatively simple: place multilayers on an electroform mandrel, electroform over it, and remove the electroformed mirror with the multilayers intact on the inside of the mirror. Making the concept in a cost effective manner, however, is a relatively elaborate process and is truly a model of interdisciplinary research: optical fabrication for making the mandrel, super hard/smooth coating technology to protect the mandrel surface *and to make it smoother*, chemistry and electronics to design an electroforming bath that produces stress-free electroforms, multilayer deposition technology, X-ray physics to design the multilayers and to characterize the multilayer performance, and optical profiler and atomic force microscopy surface characterization to provide a quantitative understanding of the mirror's performance versus surface quality. We have assembled this expertise and initial design studies have been made. Based on progress to date, we expect that it will be possible to fabricate viable Wolter I optics for hard X-ray astronomy within the near future.

5. REFERENCES

1. Gorenstein, P., et al., "The Hard X-ray Telescope Mission," BAAS, 27, no. 4, 1388, 1995.

2. White, N.E., et al., "The Next Generation X-ray Observatory," Proc. SPIE, 2518, 59–71, 1995.

3. Joensen, K.D., Gorenstein, P., Citterio, O., Høghøj, P., Anderson, I., and Schaerpf, O., "Hard X-ray Wolter-I telescope using broad-band multilayer coatings on replica substrates: problems and solutions," Proc. SPIE, 2515, 146–161, 1995.

4. Christensen, F.E., Hornstrup, A., Westergaard, N.J., Schnopper, H.W., Wood, J., and Parker, K., "A graded d-spacing multilayer telescope for high energy X-ray astronomy," Proc. SPIE, 1546, 160–167, 1992.

5. Krieger, A.S., Blake, R.L., and Siddons, D.P., "Multilayer diffraction at 104 keV," Proc. SPIE, 1742, 324–330, 1992.

6. Altkorn, R., Chang, J., Haidle, R., Takacs, P.Z., and Ulmer, M. P., "Electroform Replication of Smooth Mirrors from Sapphire Masters," Applied Optics, 31, 5153–5154, 1992.

7. Serlemitsos, P.J., private communication, 1995.

8. Joensen, K.D., et al., "Medium-sized grazing incidence high energy X-ray telescopes employing continuously graded multilayers," Proc. SPIE, 1736, 239–248, 1992.

9. Vinogradov, A.V., and Zeldovich, B.Y., "X-ray and far UV multilayer mirrors," Applied Optics, 16, 89–94, 1977.

10. Li, D., Cutiongco, E., Chung, Y.W., Wong, M.S., and Sproul, W.D., "Review of Synthesis and Characterization of Amorphous Carbon Nitride Thin Films," Diamond Films and Technology, 5, 261-273, 1995.

SUPER MIRROR FABRICATION VIA ELECTROFORMING

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ABSTRACT

As part of a project to develop methods of placing highly reflective multilayer coatings on the inside of Wolter I mirrors, we have been pursuing a program of measuring flat mirrors. These flats have been produced and examined at various stages of the process we plan to use to fabricate multilayer coated Wolter I mirrors. The flats were measured via optical profiler, AFM, (both done at Brookhaven National Lab) and X-ray reflection (done at the Argonne National Lab (ANL) Advanced Photon Source (APS)). We report for the first time, to our knowledge, the successful placement of multilayers on an electroform by depositing the multilayers on a master and then electroforming onto this master and removing the multilayers, intact, on the electroform. This process is the one we plan to use to place multilayers on the inside of Wolter I optics.

KEY WORDS X rays, Multilayers, AFM, Profiler, Wolter I

1. INTRODUCTION

In a separate paper[1], we have reported a process we are developing to produce Wolter I X-ray mirrors coated with multilayers. As part of this program we have fabricated several test flats. We measured these with a Micromap 512 profiler (simply referred to as Micromap below), an

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atomic force microscope (AFM), and with an X-ray beam at the Argonne National Laboratory Advanced Photon Source. Here we report the results of our work.

The ultimate goal of our research is to perfect the fabrication of Wolter I optics with multilayers for as low a price as possible. This will allow us to produce cost-effective X-ray optics for both astrophysical and laboratory uses. The results we report here, however, are simply to demonstrate a proof of concept of the technology by demonstrating that electroforming can be used to produce the desired optics. *No attempt was made to optimize the performance of the samples.*

2. SAMPLE DESCRIPTION

Because we were only performing the simplest proof of concept study, we used flats that were left over from previous projects. As such this set of sample is not homogenous, but the result was that we explored a range of substrates and surface qualities. We had 2 aluminum substrates: one 2-inch diameter 0.5-inch thick, the other 1-inch diameter 0.25-inch thick. These were coated with about 5 mils of electroless nickel and initially polished to sub 1 nm roughness. These samples had an additional hard protective coating made up of a 200 nm thick, approximately 10 nm period multilayer of ZrN and amorphous CN_x followed by about 20 nm of CN_x. Then they were over coated with Si/W X-ray multilayers. We also used 2 ceramic pieces about 2 mm thick that we over coated with vacuum vapor deposited gold and then coated with X-ray multilayers. Finally we coated a 1.5-inch electroformed flat that is about 1 mm thick with X-ray multilayers. These multilayers were all 100 periods of W/Si with W being the initial layer and Si the final layer. The prescribed thickness of each W layer and each Si layer was 3nm.

The X-ray multilayers described above were deposited by Atkinson Thin Film Systems of Hudson, NH. The deposition technique was DC magnetron sputtering in a 4 mtorr atmosphere of argon. The temperature of the substrates was kept below 100 C and the samples were translated between apertures in front of the Si and W sputtering targets.

Just prior to the submission of this for publication, we were able to make X-ray measurements on one more sample. This sample was made by first depositing about 40 nm of gold on top of a 2 inch diameter, 2 mm thick sapphire disk. Then 40 periods of about 1.3 nm of W and 1.3 nm of C were deposited on top. These were made at ANL, the elements were DC sputtered, the sample (Au plus sapphire) was kept a room temperature and the argon atmosphere was kept at about 2.6 mtorr. Then, an adhesion layer of Cr and Cu was evaporated. Each layer was about 50 nm thick. Finally this sapphire, Au/W/C/Cr/Cu piece was placed in an electroforming tank for about 1 day and Ni was electroformed onto the Cu surface. Then the sample was removed from the bath and the electroform was removed from the sapphire. Visual inspection showed that all the layers successfully released from the sapphire to produce proof of concept of making Wolter I mirrors in this fashion. The X-ray measurements of this sample are reported below.

3. X-ray MEASUREMENTS

3.1. GENERAL DESCRIPTION

We used an upstream mirror to act as low pass filter to prevent energies much higher than about 33 keV from reaching the sample, which is mounted on a Huber diffractometer. The distance between the upstream slit and the Huber stage that held the samples was about 5 m and the distance from the sample on the Huber to the detector on the Huber two-theta arm was about 0.8 m. The detector was NaI. The Si 111 crystals in the double crystal monochromator were deliberately tuned to reflect both lines at 10 keV (first order) and at 30 keV (third order). Two distinct voltage windows were set up to count separately the pulses from the 10 and 30 keV photons. The 10 keV data were collected mainly as a cross check that the correct energy dependence for reflectivity was observed. Once we determined that we understood the response of the mirror and the detectors, we placed a filter in the system that blocked the most of 10 keV flux. We therefore only discuss 30 keV measurements, except for the one sample (see below) that was created and tested just as this article was being completed.

The sample and axis of rotation were centered on the X-ray beam. We verified in each case that this was done correctly as the fall on of specular reflection with angle occurred at the correct absolute angle to within about 0.02 degrees.

We made two types of measurements on the samples: "theta/two-theta" scans that rotate the detector twice as far as the sample rotates relative to the beam. Alternatively we set the mirror angle to reflect at a multilayer peak and then scanned the "two-theta" arm to measure the angular spread of the reflected beam. The height of the X-ray beam was defined by a slit down stream from the monochromator. The height was about 100 microns high and about 1 mm wide. The detector slit was set at 1 mm wide for the theta-two theta scans so as to measure the integrated reflected beam and the detector slit was set to 100 mirrors for the angular spread measurements.

Below we present a plot of a few of the samples that we measured. The most significant results are the measurements made on our most recent sample. This sample was the one made via the intact removal of multilayers onto an electroform.

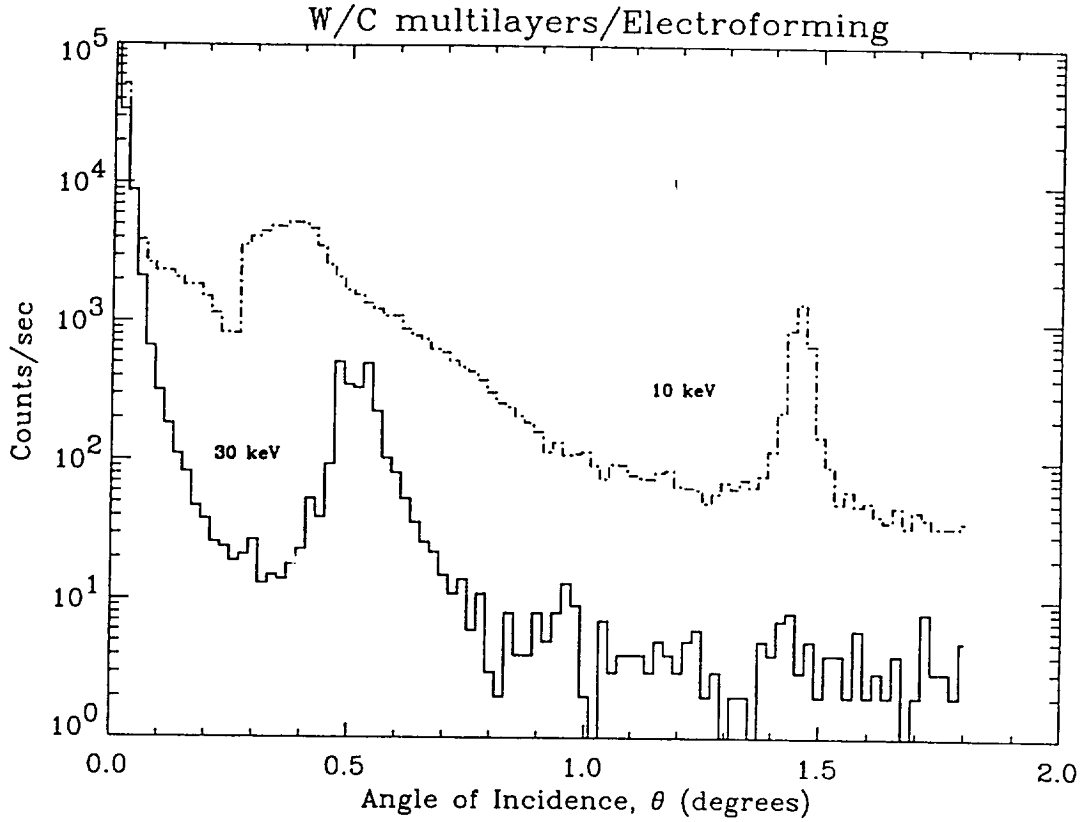

Figure 1: A theta-two theta scan of the sample that we made via electroforming onto multilayers and then removing the multilayer with the electroform.

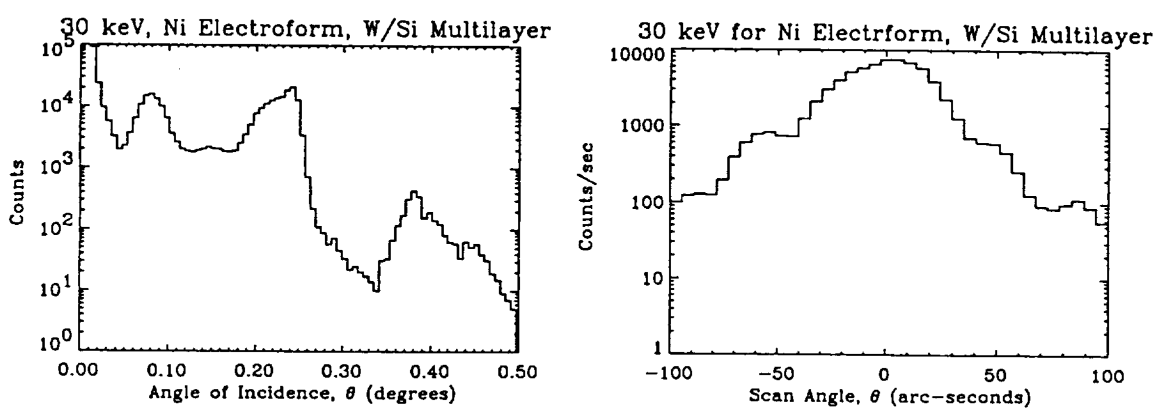

Figure 2: Left: theta/two-theta scan of an electroformed nickel "flat" (nearly flat) upon which multilayers were deposited. Right: A scan of the reflected beam with the mirror set to the angle of the multilayer peak at about 0.25 degrees seen in the figure on the left.

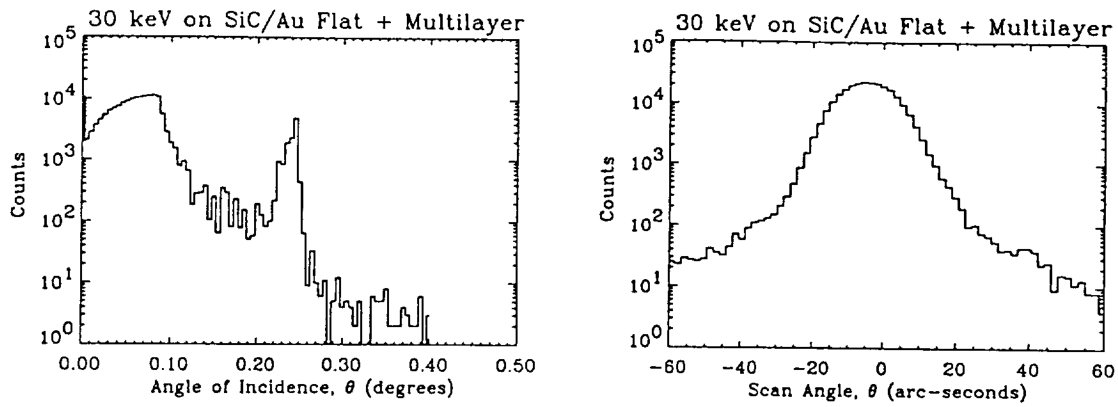

Figure 3: Left: theta/two-theta scan of an SiC "flat" (nearly flat) upon which multilayers were deposited. Right: A scan of the reflected beam with the mirror set to the angle of the multilayer peak about 0.25 degrees seen in the figure on the left.

4. DISCUSSION

4.1. X-ray results

The most important and new aspects of this work are the X-ray results shows in Figure 1 and how they relate to the prospects for producing Wolter I optics with enhanced energy coverage, increased field of view, and reduced focal length by the application of multilayers.

The X-ray results shown in Figure 1 are based on X-ray reflectivity from our sample that was made by the intact removal of multilayers onto an electroform. In Figure 1 we see two multilayer peaks, one at about 0.5 degrees for 30 keV and the other at 10 keV at about 1.5 degrees. These are consistent with a model of 40 layers of W/C with total layer thickness of 2.45 nm and a gamma of 0.4, which is close to the initial request of d = 3 nm and gamma = 0.5.

The peak in the 10 keV scan near 0.35 degrees is not yet completely understood. There are so many different material layers in this sample, however, that it is entirely possible that some combination of these layers is giving rise to the "interference effect" or peak seen near 0.35 degrees. For example, there are several other interfaces: an Au/W interface at the top of the piece; a W/Cr interface at the bottom, a Cr/Cu at the bottom; and, a Cu/Ni interface at the bottom. Some preliminary modeling of the 10 keV reflectivity by different single bi-layer combinations indicates that it is possible to obtain a feature for 10 keV reflectivity similar to that seen near 0.35 degrees in Figure 1. And, it is exciting to notice that there is the prospect for enhancing the reflectivity of mirrors at 10 keV all the way out to 1.5 degrees. Similarly, the 30 keV peak demonstrates the possibility of obtaining an increase in factor of 5 in angle over the putative 0.1 degree critical angle for a bare (no multilayers) Au surface.

In Figure 2 which is the result of multilayers evaporated onto an electroform, we see two multilayer peaks at about 0.25 (primary) and about 0.38 (first harmonic) degrees. For comparison we show results from our best flat piece in Figure 3. Here we see one multilayer peak at about 0.25 degrees. The positions of these peaks are consistent with a model in which there are 100 multilayers, 6 nm deep with a gamma of about 0.45. This is close enough to what was requested for the multilayer deposition (gamma=0.5, total size of one period, W/Si inclusive, of 6 nm) as to be quite reasonable.

Evaporation of multilayers on electroforms was not expected to be too difficult as others[2] have demonstrated that it was possible to deposit multilayers on epoxy coated aluminum foils and multilayers had been deposited once before on one of our electroforms[3]. But a direct deposition technique does not allow the fabrication of multilayer coatings on the inside of Wolter I optics unless the optics are about a meter or more in diameter to allow for the uniform deposition of the multilayers. Or, if smaller mirror diameters are to be combined with direct multilayer deposition, then the mirror must be only a fraction of a complete conic of revolution. Usually about one quarter of the complete 360 degrees of revolution is used[4,5]. Then these "shells" will need to be assembled into a pseudo-Wolter I optic[4,5]. Thus, our demonstration that it is possible to remove multilayers from a master onto a Ni via electroforming is real breakthrough in technique, as this means that it will be possible to produce relatively small and low cost Wolter I (or optics of similar geometry) X-ray super mirrors.

4.2. NON X-RAY EVALUATION

In order to fabricate high performance X-ray optics, quality control is important and related to this aspect of X-ray mirror fabrication we discuss in this section results of the surface quality as measured via the Micromap and AFM, and how they predict the performance of the X-ray results. We first begin with a summary of the Micromap (and in two cases AFM) measurements.

All the substrates were measured prior to being coated with multilayers. Time did not permit us make Micromap measurements after the multilayer deposition. In addition to the Micromap, AFM measurements were made only on the 2 aluminum/electroless nickel substrates. For these two pieces, Micromap results differ significantly, but AFM results do not. Below we discuss the comparison of the X-ray measurements with the Micromap measurements. In Table 1 below we provide a summary of the Micromap measurements on our samples. Micromap measurements were made before and after the CN_xZrN coated Ni/Al samples were over coated with about 50 nm of Ni (to be used as a release agent). Thus the Ni/Al pieces are listed twice in Table 1.

Table 1: Summary of Micromap Measurements. Contour is the root mean square deviation from on a surface contour, and Ra is the RMS of the deviations from a two dimensional profile from the average plane. Both numbers are based on averaging over a 3.25 mm by 0.8 mm area. A 2.5 x objective was used. To describe the samples, we list the elements (or materials), from right to left, starting from the surface and ending with the bottom substrate.

Sample	Contour	Ra
CN_x/ZrN/Ni/Al, 1″ dia.	0.97 nm	0.71 nm
Ni/CN_x/ZrN/Ni/Al, 1″ dia.	1.07 nm	0.81 nm
CN_x/ZrN/Ni/Ni/Al, 2″ dia.	1.07 nm	0.82 nm
Ni/CN_x/ZrN/Ni/Al, 2″ dia.	1.21 nm	0.84 nm
Au, sapphire, 2″ dia	0.27 nm	0.28 nm
Au, CVD SiC 1″ sq.	0.45 nm	0.31 nm
Au, Ni electrofm., 1.5″ dia.	0.79 nm	0.63 nm

In a previous SPIE proceeding it was suggested[6] that AFM measurements were necessary and sufficient to predict the X-ray reflectivity characteristics of X-ray mirrors. This previous work also reported WYKO (another brand of optical profiler) measurements made with a 20 x objective and the PDS of the WYKO was indeed quite disparate from the AFM PDS (c.f. their Figure 6). In contrast, our Figure 4 (shown below)

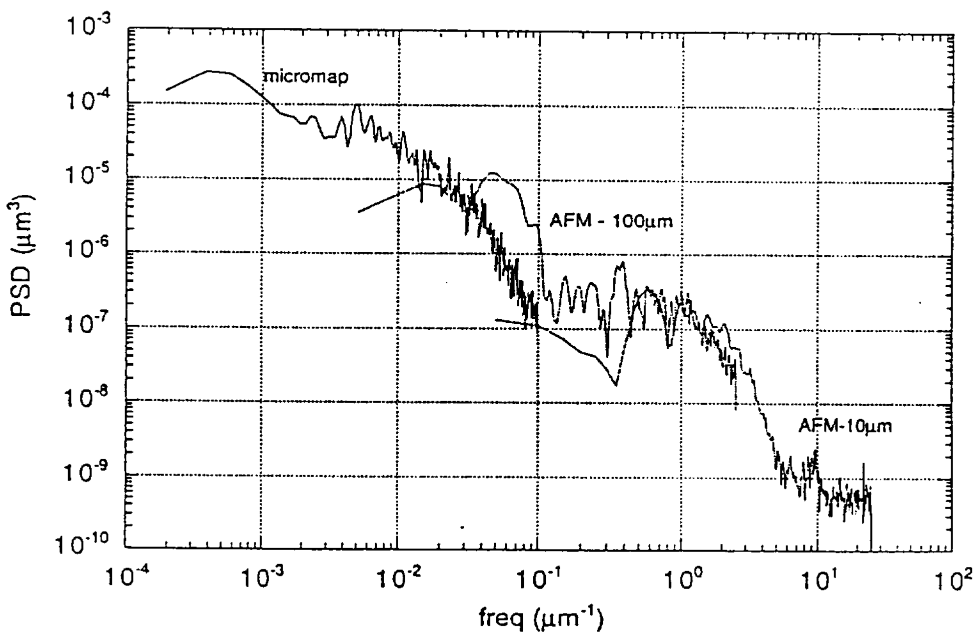

Figure 4: Micromap measurements and AFM measurement of sample the 2 in. nickel sample in Table 1.

with a restored PDS shows how the Micromap data give better coverage and comparable amplitudes to those of the AFM in the critical 5×10^{-2} to $5 \times 10^{-1}(m\mu)^{-1}$ frequency domain. We suspect the reason for the disparate AFM and WYKO results in this previous work[2] was because for the WYKO results, they did not use a restored PDS. We suggest, therefore that Micromap measurements are valid for evaluating the surface quality of X-ray mirrors and we provide further evidence for this below.

With multilayers added to the surface, there is an extra requirement on surface smoothness besides the standard grating equation and scattering dependence of surface quality. This is that the quality of the multilayers is generally affected by the surface upon which they are deposited, though at least one group[2] suggests that it is possible to produce effective ion polishing of the substrate by polishing the intial layers of the multilayers with Kr^+ ions. And another group[7] suggests that certain multilayer combinations are self-planarizing. No special treatment was applied to our multilayers, however, and comparison between X-ray measurements and the Micromap results can be used to judge how the quality of our multilayers was affected by the the substrate smoothness.

Time did not permit detailed calculations based on the Micromap results to compare with our X-ray measurements. We did, however, perform two comparisons between the X-ray results and the Micromap measurements which demonstrates a correlation between the average Micromap measurements (c.f. Table 1) and the X-ray performance of multilayers. Both techniques are susceptible to errors in the macro figure error and alignment with the X-ray beam so that the correlations could be masked by systematic effects. The Micromap measurements of those pieces listed in Table 1 indicate, however, that the radii of curvature of these flats were large. They ranged from kilometers to tens of meters as measured over mm portions of the flats. And, the general trend in Figure 5 is certainly consistent with the hypothesis that the Micromap measurements are sufficient for predicting the behavior of multilayers.

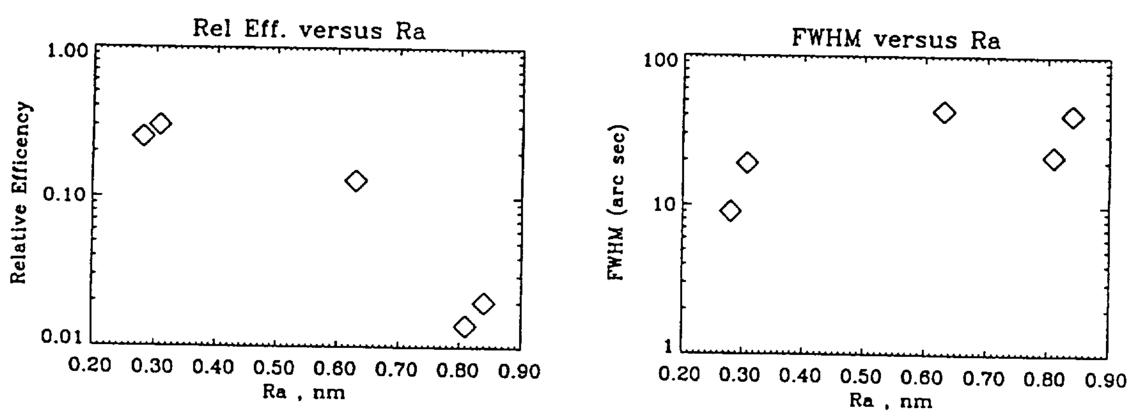

Figure 5: Left: Plot of the relative efficiency of the multilayer peak as defined by the ratio the total counts of the reflected beam at the multilayer peak (c.f. Figures 1 and 2, right hand panels) and the direct beam versus the Ra average values measured for the samples (c.f. Table 1 for Ra values). Right, FWHM of the same multilayer peak versus the Ra of the samples.

5. SUMMARY AND CONCLUSION

We have shown, for the first time, that it is possible to remove multilayers intact from a master onto an electroform and we conclude that the outlook is bright for applying this technique to the fabrication Wolter I super mirrors.

We also find that it is possible to obtain good performance from multilayers deposited on ceramics as well metal surfaces including both originally polished ones and one produced via electroforming replication.

In order to quality control the process, we find that the Micromap profiler measurements on the substrate prior to the deposition of the multilayers are good predictors of the performance of the multilayers. The specific process we used to electroform onto a multilayered master was the simplest one we could devise, as we knew that Au releases from sapphire when we electroform on the Au sapphire combination. We used about 40 nm of gold because we knew that this amount of Au makes smooth surfaces. Much thinner depositions at room temperature (the temperature used here) could lead to the formation of 'islands" which would make the surface too rough for the effective deposition of functional multilayers.

We are now in the process of perfecting the deposition of smoother release layers that are less absorbing. We will include both Au on a heated substrate, Ni, and other materials. We also will advance this technique to metal masters that have been coated with a smoothing and protecting agent such as CN_x[1].

Further improvements to the technique we plan to make are to improve the surface quality upon which the multilayers are deposited and to improve the quality control and handling of the electroformed pieces. When all these improvements are made, we fully expect to achieve angular resolution below 1 arc minute and we do not rule out resolution in the arc second range.

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7. REFERENCES

1. Ulmer, M. P., Altkorn, R., Krieger, A., Parsignault, D., Chung, Y.-W., "Grazing incidence and multilayer X-ray optical systems," Proc. SPIE,3113, 1997 to be published

2. Hussain, A. M., Joesen, K. D., Høghøj, Christensen, F. E., Louis, E., Voorma, H. J., Soong, Y., White, N., Serlemitsos, P. J., and Anderson, I., "The Deposition and Characterization of Multilayers on the Thin Foil X-ray Mirrors for High Throughput X-ray telescopes," Proc. SPIE, 2805, 336–342, 1996.

3. Ulmer, M. P., Altkorn, R., Hailde, R., Madsen, D. "Production of Ultrasmooth Electroform-Replica Mirrors for Multilayer Optics," Proc SPIE, 2011, 100-106, 1994.

4. White, N.E., et al., "The Next Generation X-ray Observatory," Proc. SPIE, 2518, 59–71, 1995.

5. Christensen, F.E., Hornstrup, A., Westergaard, N.J., Schnopper, H.W., Wood, J., and Parker, K., "A graded d-spacing multilayer telescope for high energy X-ray astronomy," Proc. SPIE, 1546, 160–167, 1992.

6. Clark, A. M., Bruni, R. J, Romaine, S. E. , Schwartz, D. A., Van Speybroeck, L., Yip, P. W., Drehman, A. J., and Shapiro, A. P.," Correlation between X-ray reflectivity measurements and surface roughness of AXAF coated witness samples," Proc. SPIE, 2805, 268–276, 1996

7. Thomas, C., Ulmer, M. P., and Ketterson, J. B, "Superconducting Tunnel Junction Base Electrode Planarization," submitted to Applied Physics letters, 1997.

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This document is the final technical report for contract NASW-96008, "Development of a Focussing Hard X-ray Telescope". The period of performance of the contract was from June 17, 1996 to June 16, 1997. This contract was the most recent phase of an ongoing program at Radiation Science supported by NASA. The overall objective of this program is the construction of a focussing hard X-ray telescope.

We were able to generate replicated surfaces with arc-second range hard X-ray scattering properties. We were able to reflect 15, 30, and 45 keV X-rays from multilayer coatings on thin foils. We were able to separate a multilayer coating deposited directly on a master surface from the surface after electroforming a supporting structure without damaging either the multilayer or the master surface. These developments open the door for the fabrication of a Wolter type I hard X-ray telescope. We conclude that the development of such a telescope should proceed.

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